A novel methodology to determine the mechanical properties of amorphous materials through instrumented nanoindentation

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SUMMARY

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   - Objectives

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5. APPLICATION

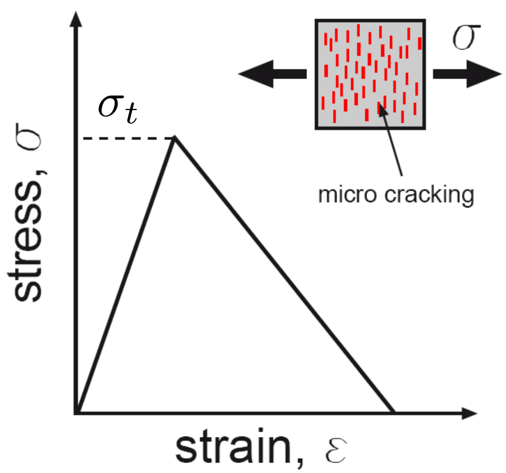
6. CONCLUSIONS

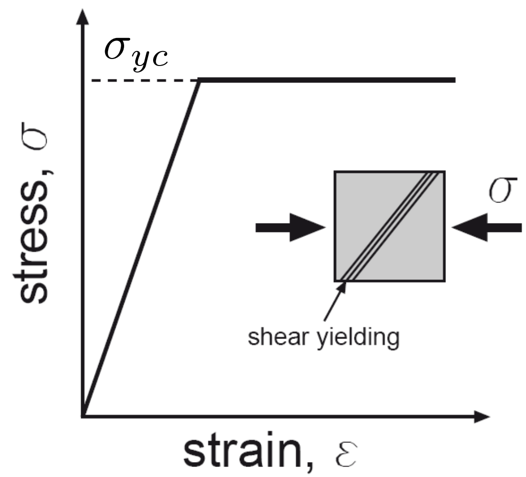
MECHANICAL BEHAVIOR of AMORPHOUS SOLIDS

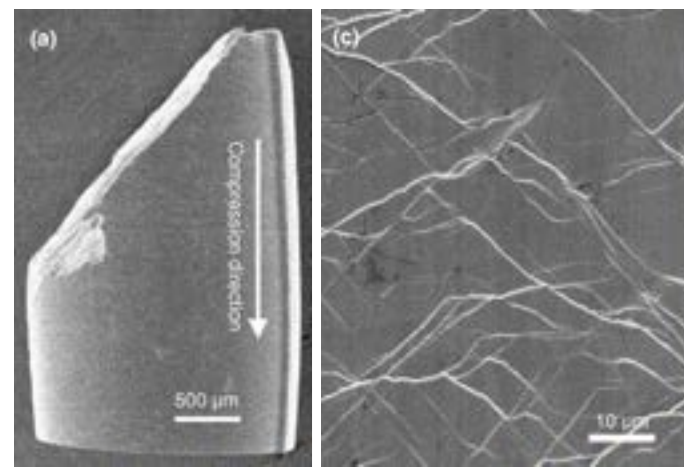
- Mechanical behavior of amorphous solids (ceramic glasses, metallic glasses and thermoset/thermoplastic polymers) presents a number of similar features:

Brittle in tension

Ductile in compression/shear by the propagation of shear bands
Yield strength is pressure-sensitive
Limited strain hardening







- Determination of the mechanical properties by standard tests is sometimes difficult (brittle behavior in tension).
- Instrumented nanoindentation emerges as an alternative with the additional advantage of in situ testing.

- Mechanical behavior of amorphous solids is well represented by the Drucker-Prager model (rate effects are neglected):

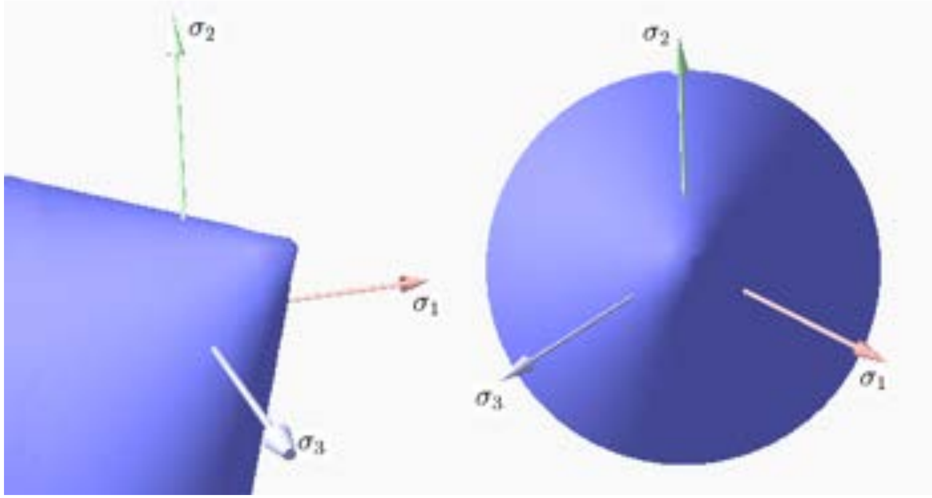
Drucker-Prager yield surface

$$\Phi = \sqrt{3J_2} - d + \frac{I_1}{3} \tan\beta = 0$$

$$I_1 = \sigma_{ii} \qquad \sigma'_{ij} = \sigma_{ij} - I_1/3$$

$$J_2 = \tfrac{1}{2}\sigma'_{ij}\sigma'_{ji}$$

$$\frac{\sigma_{yc}}{\sigma_t} = 1 + \tan\beta$$



- Mechanical properties of amorphous solids are determined by: (rate effects are neglected):

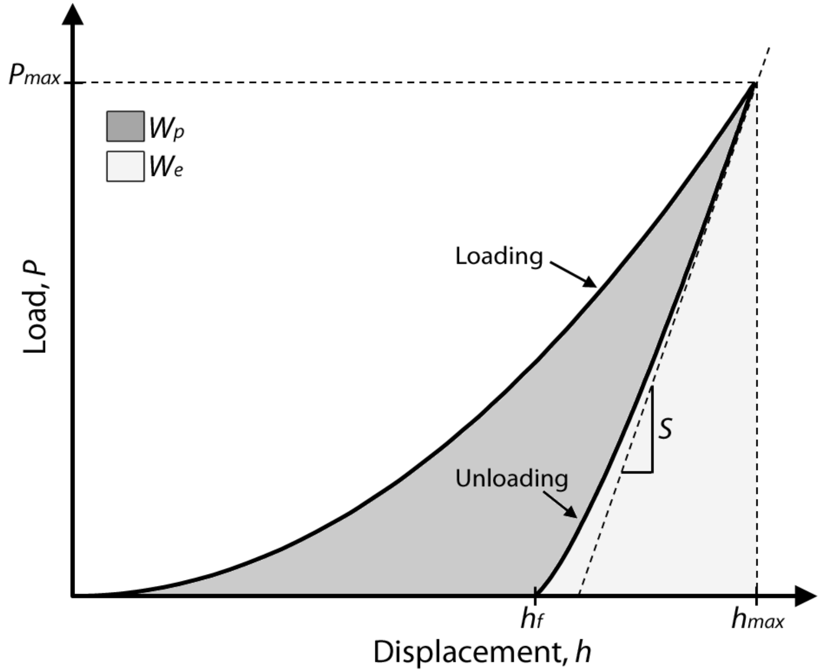
Cohesion: d

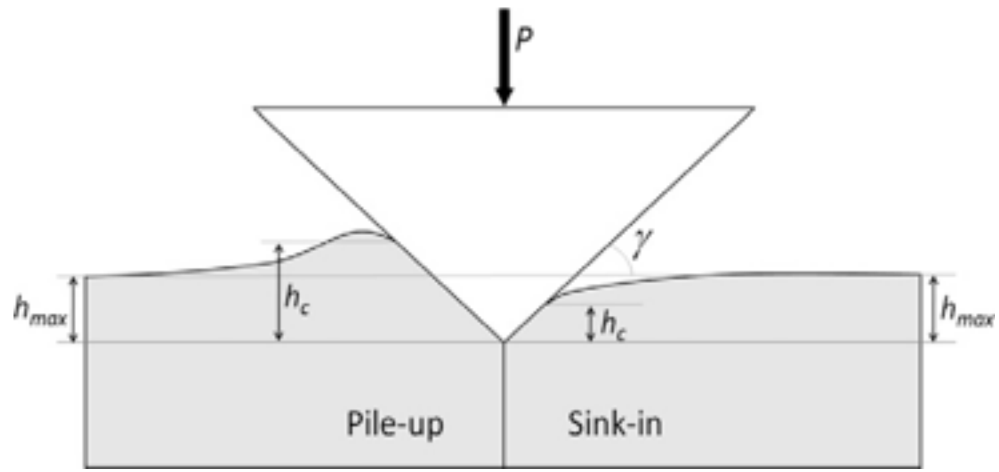
Friction angle: β

Elastic constants: $E,\ \nu$

- Can they be obtained from instrumented indentation tests?

INSTRUMENTED INDENTATION TEST





- Four parameters:

$$P_{max},\ h_{max},\ S,\ W_p/(W_e+W_p)$$

but only two are independent ...

- Uncertainty in the contact area

$$H_{ap} = \frac{P_{max}}{A_{ap}} \qquad c_p = \sqrt{A/A_{ap}}$$

c_p is given by analytical expressions (Oliver & Pharr, 1992)

- This information is used to compute the hardness and the elastic modulus

$$H = \frac{P_{max}}{A}$$

$$E^* = \frac{S\sqrt{\pi}}{2\sqrt{A}} \qquad \left(\frac{1}{E^*} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}\right)$$

INVERSE PROBLEM

The inverse problem (determination of yield stress from hardness) is not trivial in general because different combinations of material properties (yield stress, pressure sensitivity, strain hardening) may lead to the same hardness.

Known results: Von Mises solid $$\frac{H}{\sigma_{yc}} = f\left(\frac{\sigma_{yc}}{E^*}\right)$$ $f \approx 3$ in the fully-plastic regime

Drucker-Prager solid: $$\frac{H}{\sigma_{yc}} = f\left(\frac{\sigma_{yc}}{E^*}, \beta\right)$$

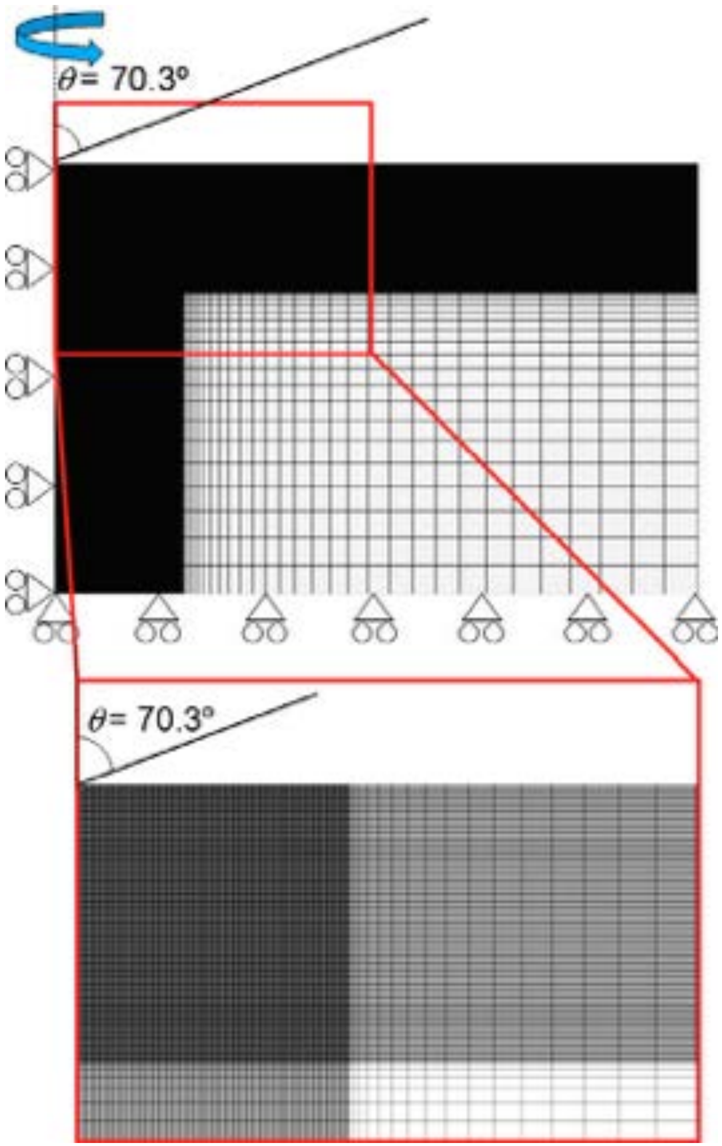
Main assumption: σ_{yc} and β are replaced by a characteristic indentation stress σ_r

$$\sigma_r = d - \sigma_h \tan\beta = d - aH\tan\beta$$

$$\frac{H}{\sigma_r} = f\left(\frac{\sigma_r}{E^*}\right) \qquad \sigma_{yc} = \frac{\sigma_r + aH\tan\beta}{1 - \tan\beta/2}$$

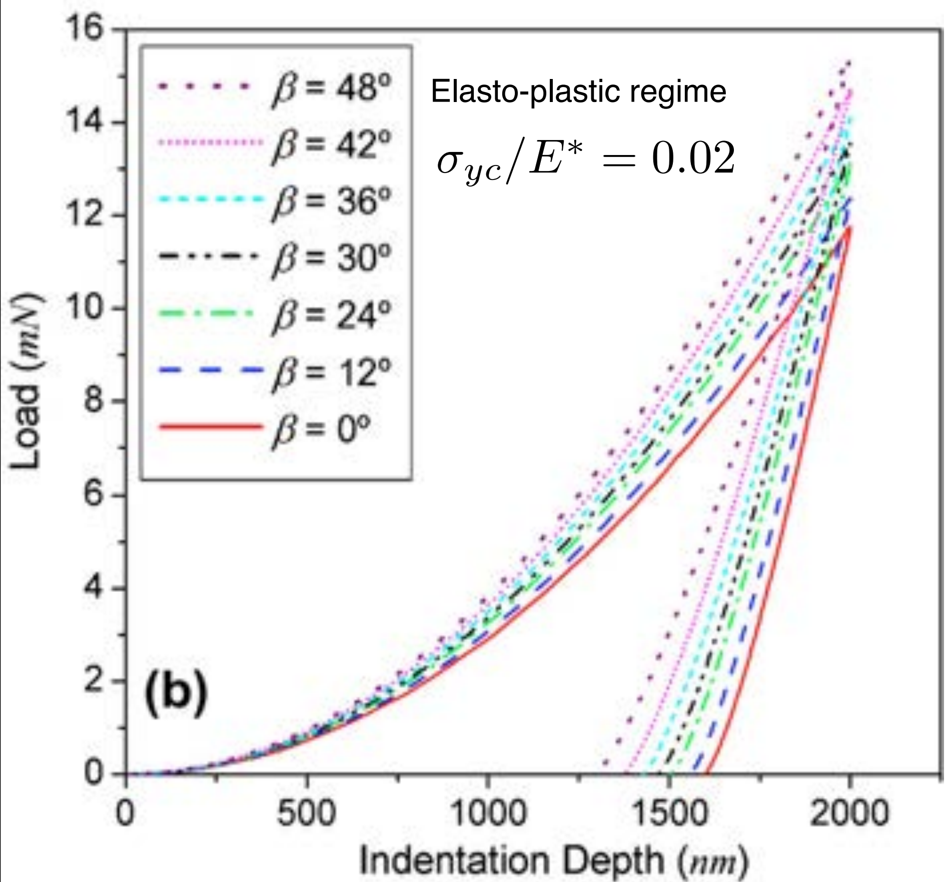
The validity of this assumption and the value of the constant a can be obtained from the finite element simulation of the indentation test in a Drucker-Prager solid

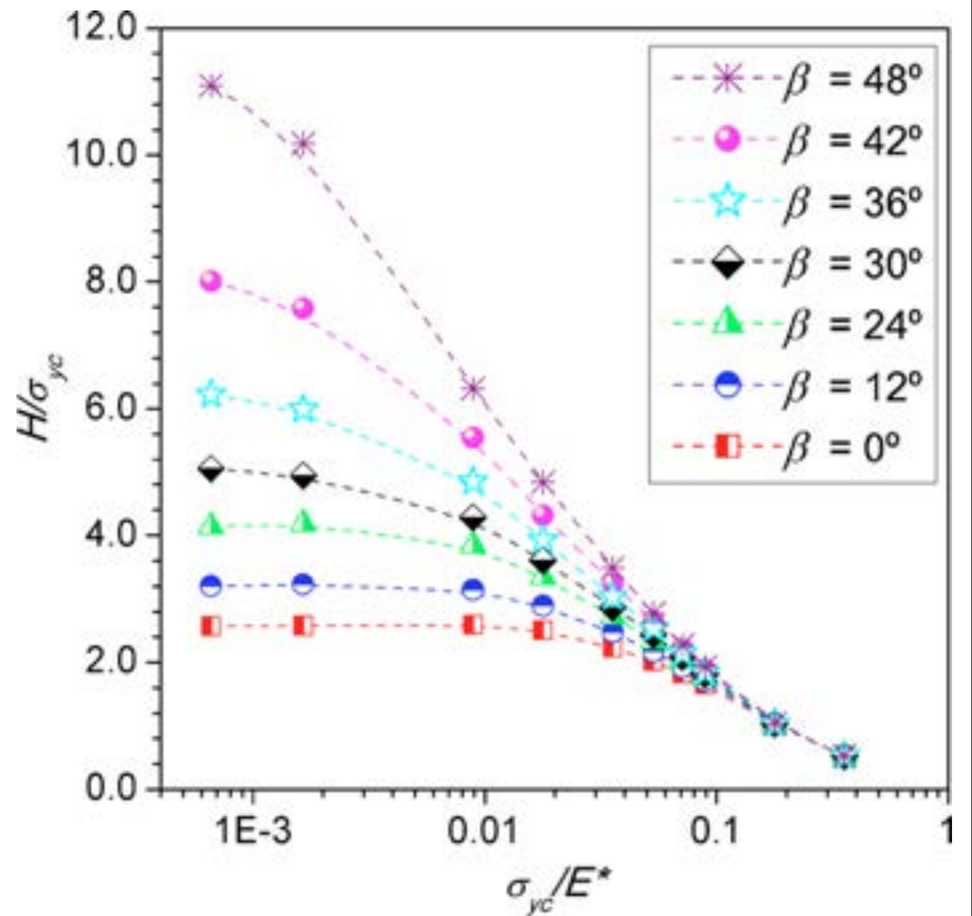
FINITE ELEMENT MODEL

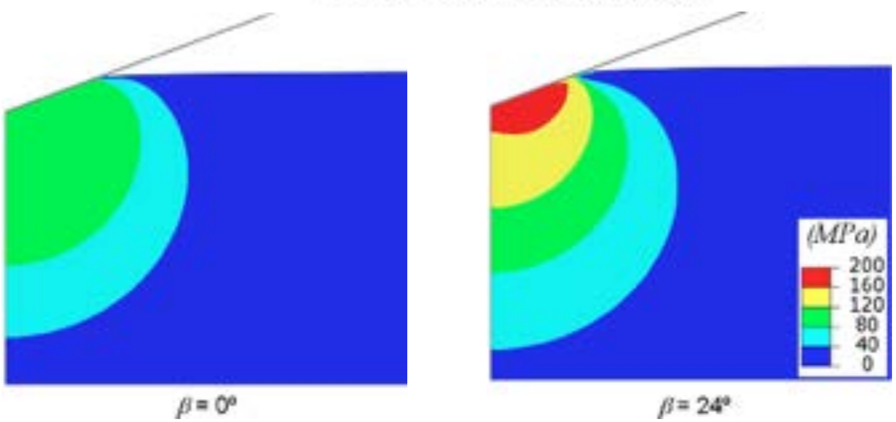


- Axisymmetric model, large deformations.
- Elasto-plastic Drucker-Prager material.
- Rigid conical indenter equivalent to a Berkovich tip (θ = 70.30º).
- Frictionless indenter/material contact.
- Discretization with first order elements with full integration (CAX4). Mesh refined at the contact area with the tip (40 nodes in contact).
- Simulations carried out with Abaqus/Standard.
- In cases of excessive distortion, simulations were carried out with Abaqus/explicit and arbitrary Lagrangian–Eulerian mesh adaptivity.

NUMERICAL RESULTS





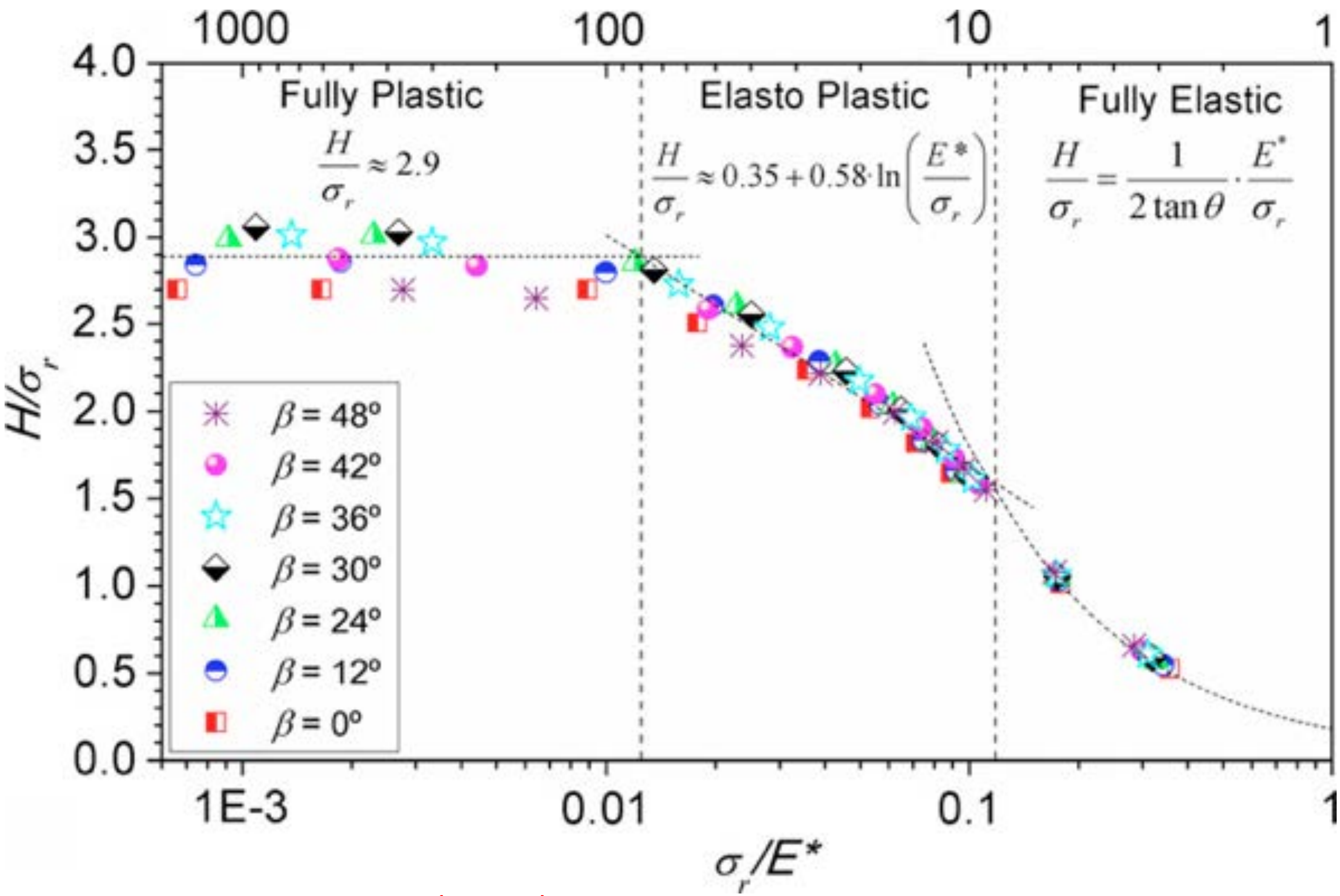


$$\frac{H}{\sigma_{yc}} = f\left(\frac{\sigma_{yc}}{E^*}, \beta\right)$$

?

$$\frac{H}{\sigma_r} = f\left(\frac{\sigma_r}{E^*}\right)$$

NUMERICAL RESULTS

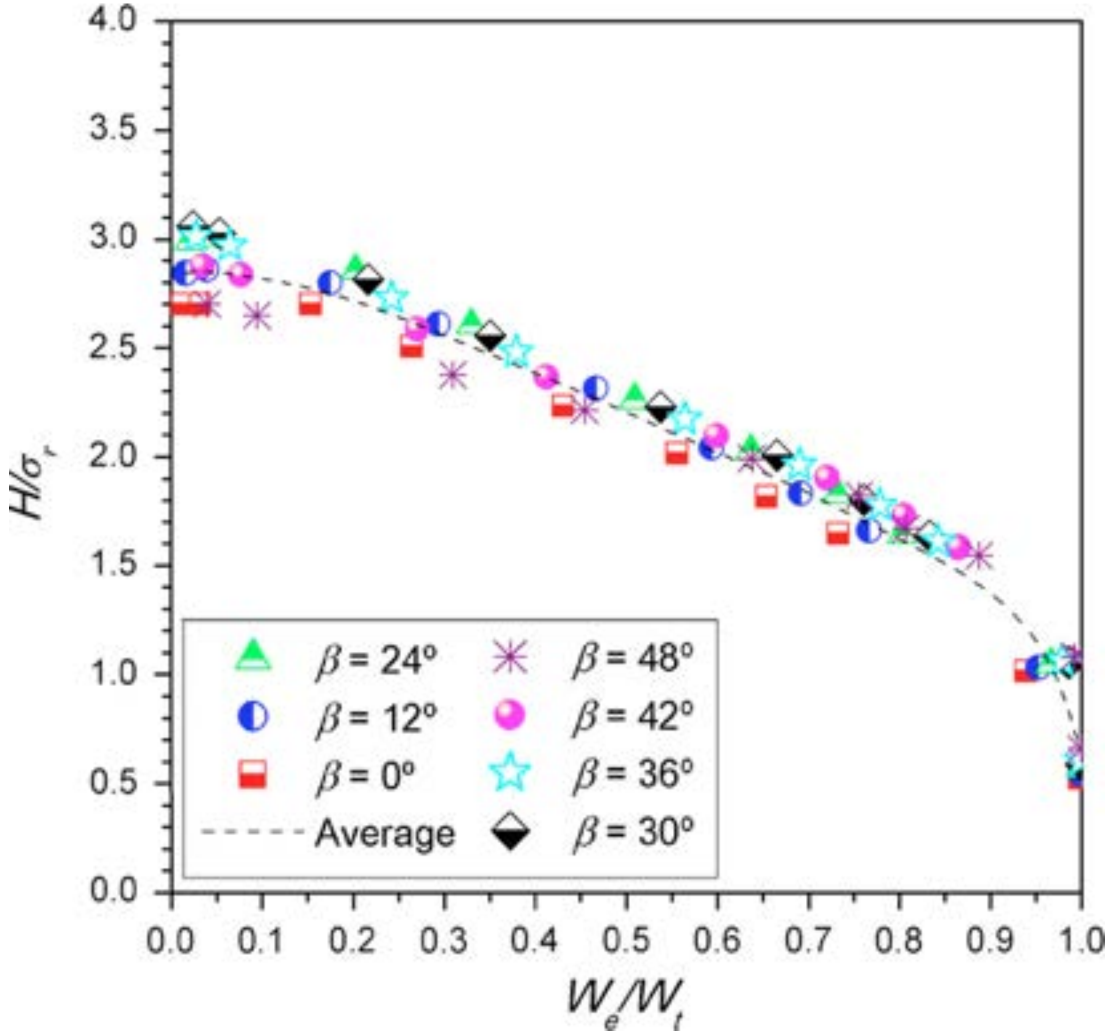


$$\frac{H}{\sigma_r} = f\left(\frac{\sigma_r}{E^*}\right)$$

$$\sigma_r = d - 0.29 H \tan\beta$$

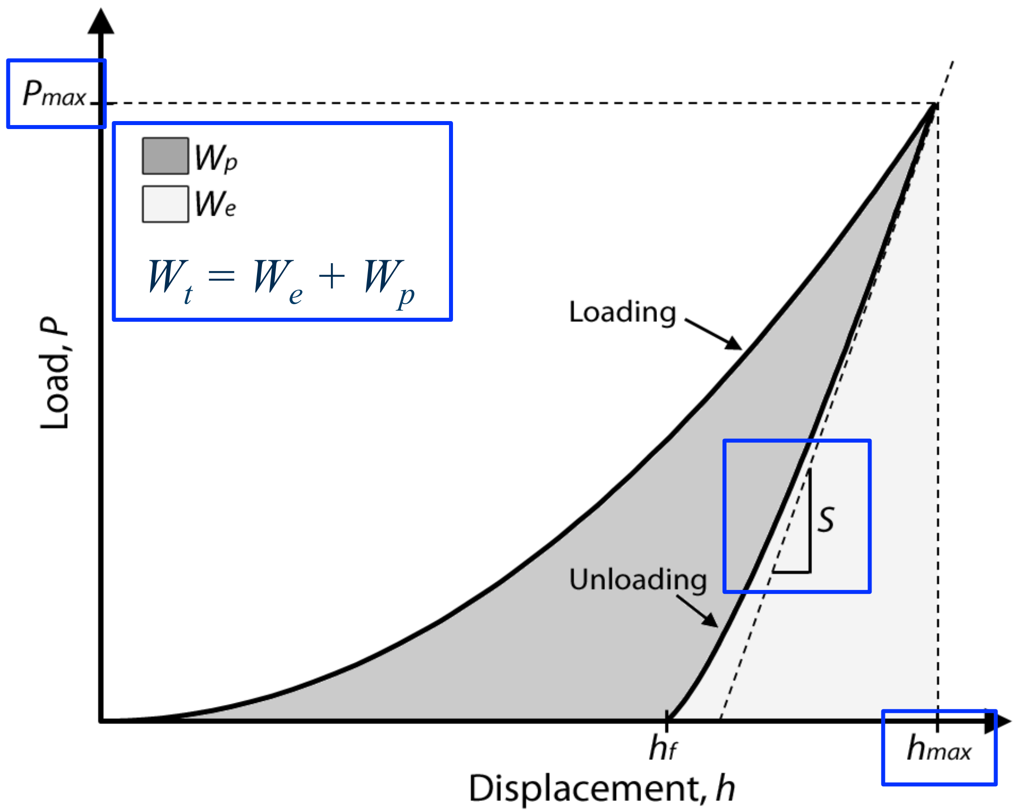
NUMERICAL RESULTS

W_e/W_t is closely related to σ_r/E^*



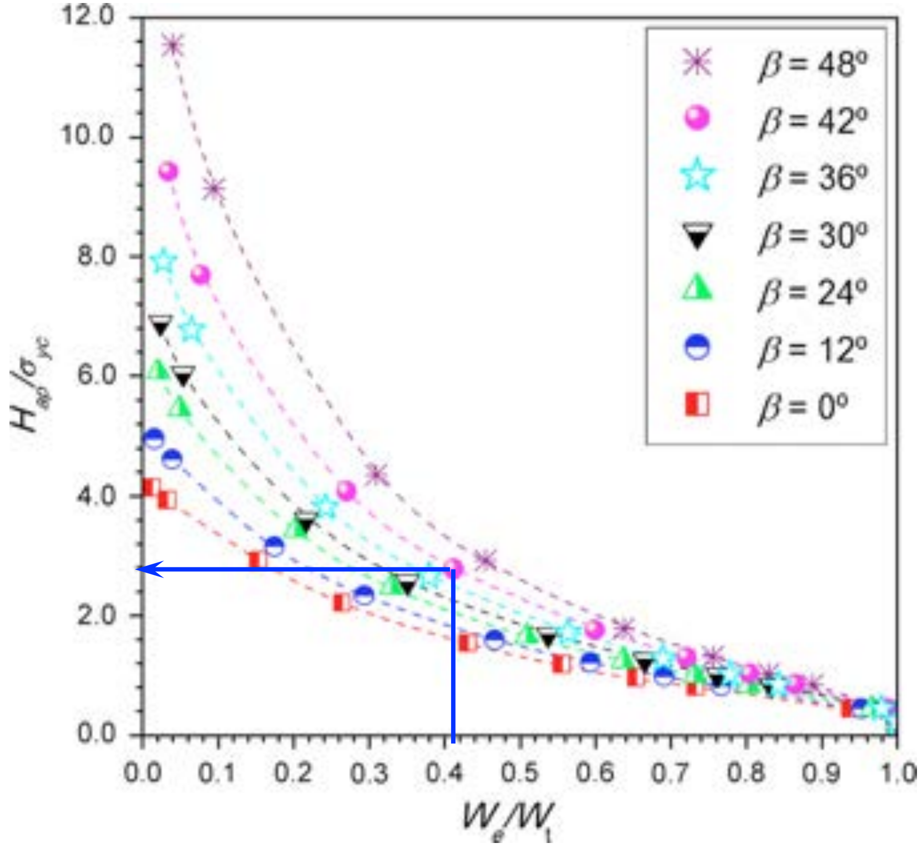
INVERSE PROBLEM

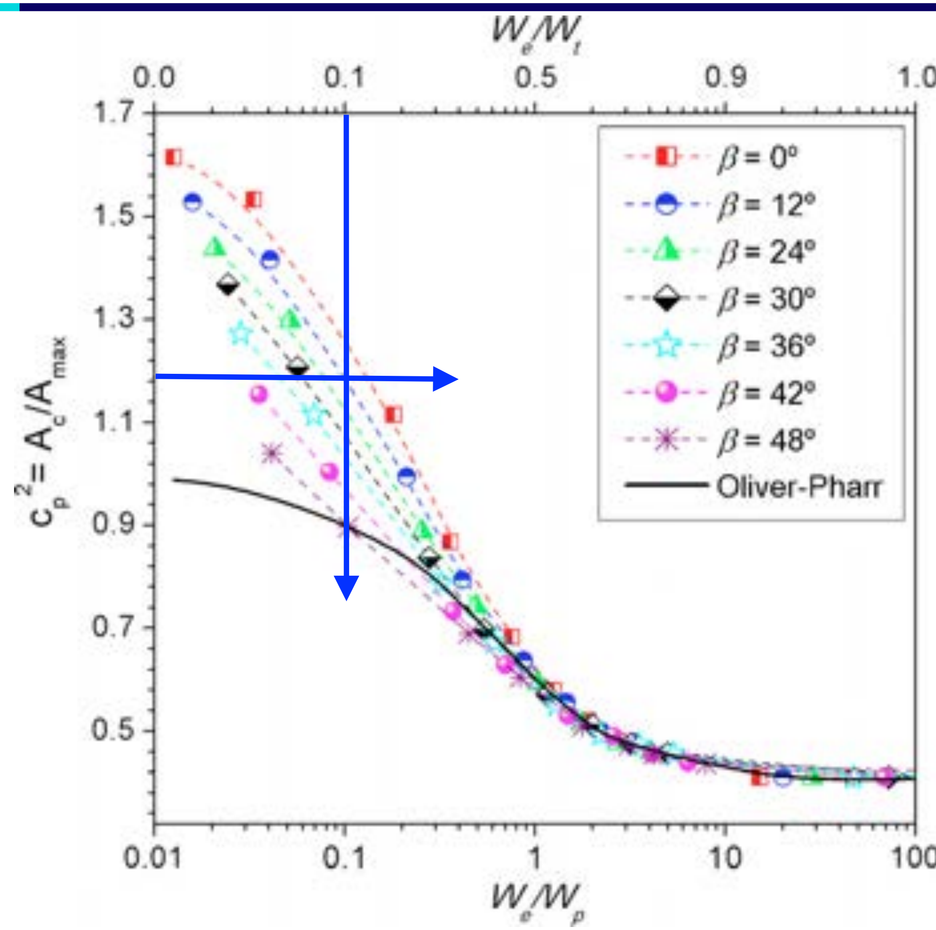
Determination of E^*, σ_{yc} and β from instrumented indentation: $H_{ap},\ S,\ W_e/W_t$



$$H_{ap} = \frac{P_{max}}{A_{ap}}$$

MASTER CURVES





- Master curves are built as a function of parameters readily measurable and incorporate the effect of the pile-up

- The master curves allow to determine σ_{yc} from the indentation data when β is known and viceversa

- In addition, it is possible to determine β from the independent measurement of the real contact area A_c if $W_e/W_t < 0.5$

MATERIALS

- Bulk metallic glasses: $Zr_{65}Cu_{15}Al_{10}Ni_{10}$, $Mg_{58.5}Cu_{30.5}Y_{11}$ and $Mg_{61}Cu_{28}Gd_{11}$
- Ceramic glasses: Soda-lime glass (Starphire®) and borosilicate glass (Borofloat®)
- Glassy polymers: epoxy, PMMA and PVD
- Wide range of σ_{yc}/E and β

EXPERIMENTAL TECHNIQUES

- Experiments conducted using a Nanoindenter XP
- At least 10 indentations in each material with Berkovich tip at a strain rate 0.002 s^{-1}
- Residual imprints measured by AFM using a Park XE150

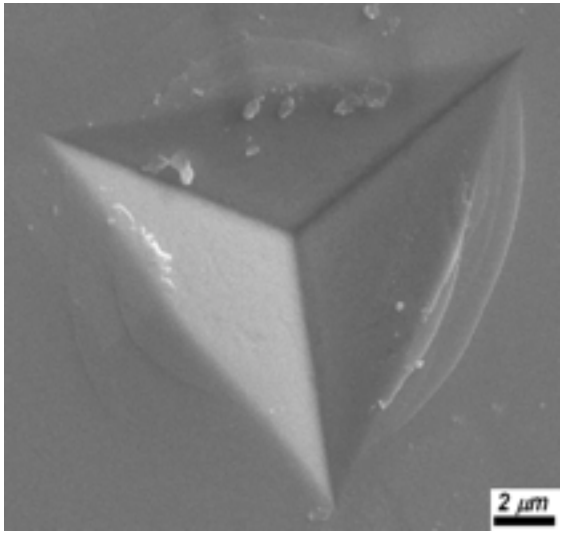
Bulk Metallic Glasses

Analysis Strategy

- $W_e/W_t \approx 0.31$
- No prior knowledge of σ_{yc} or β available

} Measurement of Residual Imprint by AFM

$$\left.\begin{matrix}A_{ap}\\ A_c\end{matrix}\right\} c_p \xrightarrow[W_e/W_t]{} \beta \xrightarrow[W_e/W_t,\; H_{ap}]{} \sigma_{yc}$$



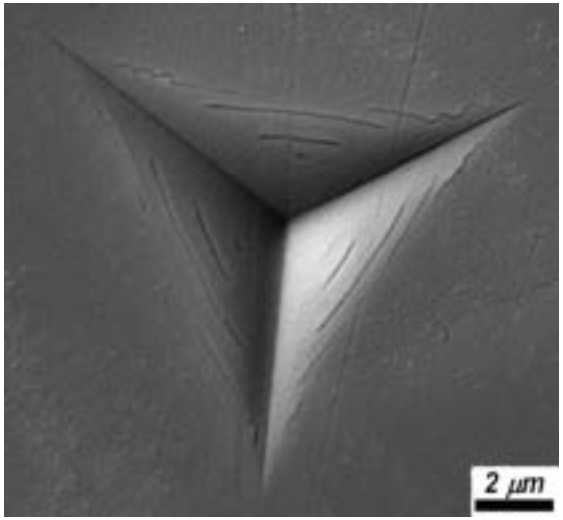
Ceramic Glasses

Analysis Strategy

- W_e/W_t in the range 0.52-0.58
- Oliver & Pharr provides A_c but β cannot be determined from A_c

} σ_{yc} determined by mechanical testing

$$W_e/W_t,\; H_{ap}/\sigma_{yc} \longrightarrow \beta$$

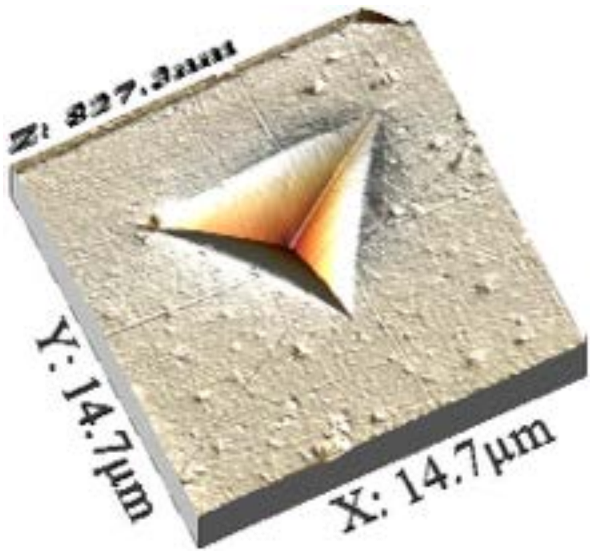


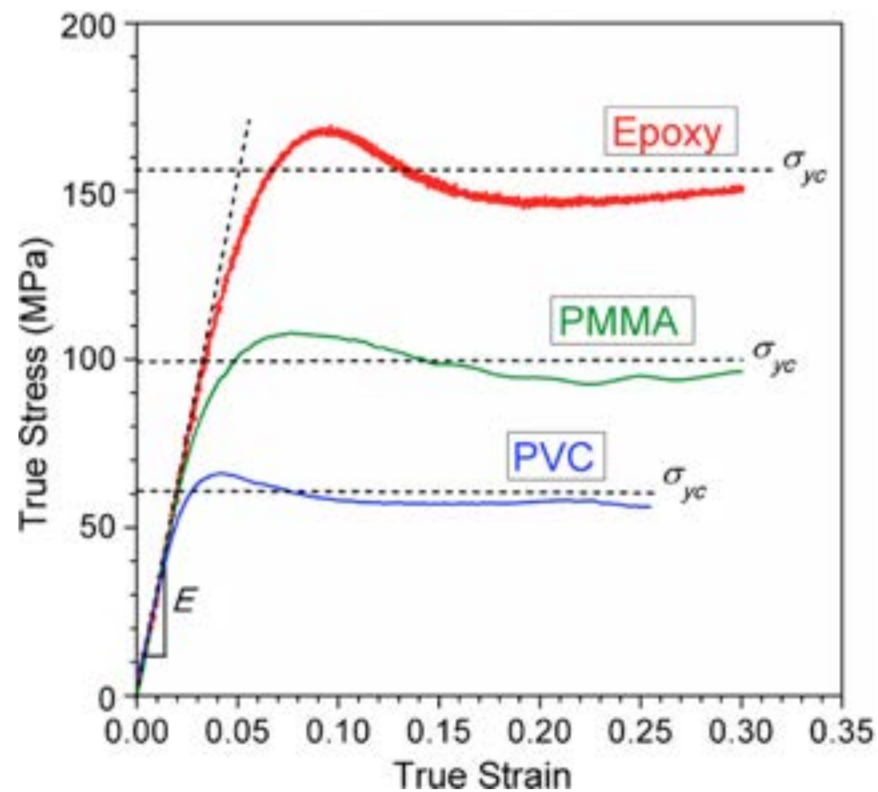
Analysis Strategy

- W_e/W_t in the range 0.31-0.58
- Residual imprint cannot be measured because of viscoelastic effects

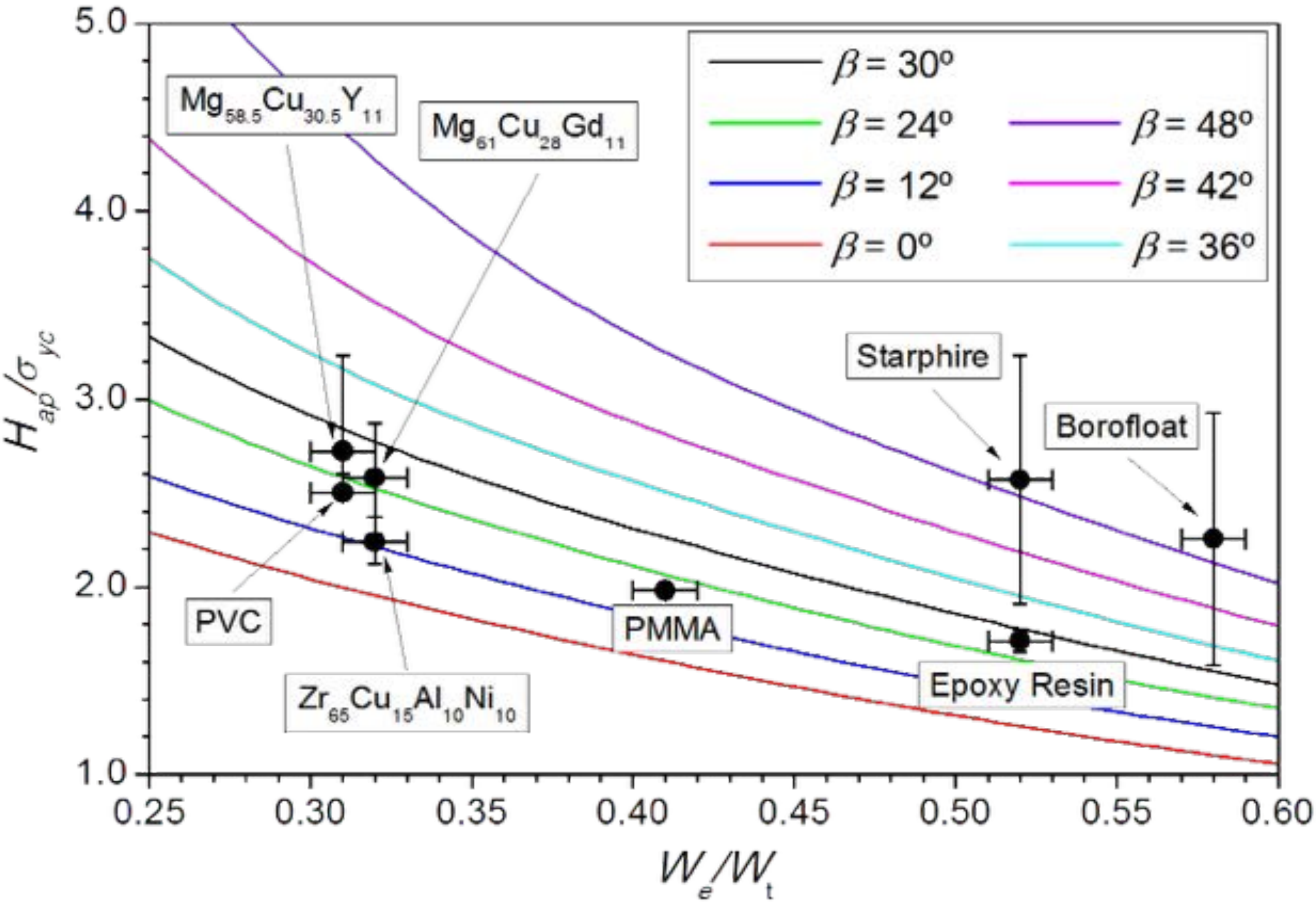
} σ_{yc} determined by mechanical testing

$$W_e/W_t\,,\ H_{ap}/\sigma_{yc} \longrightarrow \beta$$

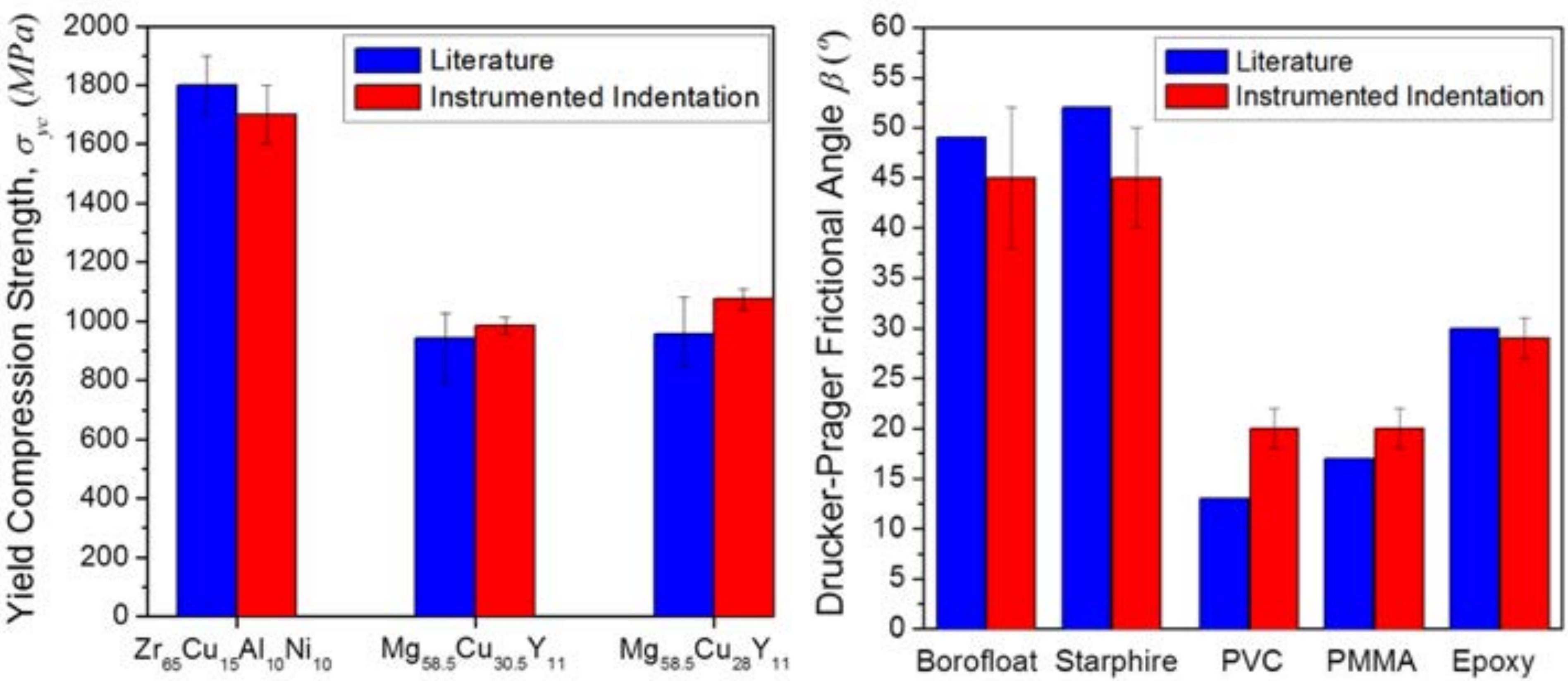




Experimental Range



Literature Comparison



Bulk Metallic Glasses

Ceramic Glasses

Glassy Polymers

CONCLUSIONS

- A novel methodology based on instrumented indentation was developed to characterize the mechanical properties (*E*, σ_{yc} or β) of amorphous materials.
- The approach is based on the concept of a universal postulate that assumes the existence of a characteristic indentation pressure proportional to the hardness. This hypothesis was numerically validated.
- This method overcomes the limitation of the conventional indentation models (pile-up effects and pressure sensitivity materials)

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Research Projects

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- Comunidad de Madrid, Program ESTRUMAT-CM
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